

m-12977



[REDACTED]
George Kovall
Mosel-Vitellic, Inc.
3910 North First Street
San Jose, CA 95134-1501, USA

Subject: XPS/ESCA Report
CEA Number: C02G3712
Purchase Order Number: 200187

Dear George:

Enclosed is the final report for the sample(s) submitted for analysis.

Date received:	[REDACTED]
Results faxed/emailed:	[REDACTED]
Results emailed to:	gkovall@mosel-vitellic.com
Number of samples:	1
Number of units:	2.50
Analysis cost (@ \$[REDACTED] per unit):	\$[REDACTED]
Priority Surcharge: (0%)	\$0.00
Total analysis cost:	\$[REDACTED]

Your sample(s) will be retained for eight weeks after their receipt. **After this time they will be disposed of, unless you specifically request otherwise.** We will maintain copies of the report and data for three years.

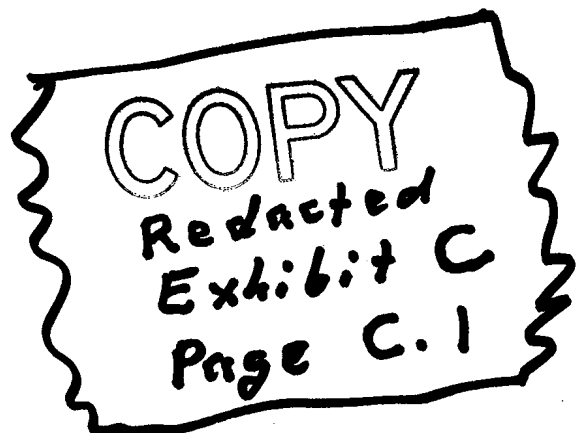
Thank you for using the analytical services of Charles Evans & Associates, a division of the Evans Analytical Group. We appreciate your business and welcome any suggestions you may have for improving the quality and efficiency of our service. Please do not hesitate to call us if you have any questions regarding this report.

Sincerely,

A handwritten signature in cursive script, appearing to read 'Thomas F. Fister'.

Thomas F. Fister, Ph.D.
Manager, XPS Services
(Tel. 408-530-3660; Email: tfister@cea.com)

Enclosures:





**X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)/
ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS (ESCA)
SURFACE ANALYSIS REPORT
CEA NUMBER C02G3712
PO NUMBER 200187**

for

George Kovall
Mosel-Vitellic, Inc.
3910 North First Street
San Jose, CA 95134-1501, USA

Prepared by:

A handwritten signature in black ink, appearing to read "Thomas F. Fister".

Thomas F. Fister, Ph.D.
Manager, XPS Services
(Tel. 408-530-3660; tfister@cea.com)

Reviewed by:

A handwritten signature in black ink, appearing to read "Greg Strossman".

Greg Strossman, Ph.D.
Specialist, XPS Services
(Tel. 408-530-3798; gstrossman@cea.com)

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A handwritten label consisting of the text "C-2" enclosed in a hand-drawn, irregular rectangular border.

XPS/ESCA ANALYSIS LABORATORY REPORT

Requester:
CEA Number:
Analysis Date:

George Kovall
C02G3712

Purpose:

The goal of this analysis was to determine the nature of a residue on a TiN substrate. The residue was found on a bare area in between a set of arrays on die V2. The residue remained following an etch process to remove an oxide/photoresist pattern.

Summary:

The residue consists of a mixture of fluorocarbons, N-containing organics, Ti fluoride and ammonium species (possibly as NH_4F).

Experimental:

XPS data is quantified using relative sensitivity factors and a model that assumes a homogeneous layer. The analysis volume is the product of the analysis area (spot size or aperture size) and the depth of information. Photoelectrons are generated within the X-ray penetration depth (typically many microns), but only the photoelectrons within the top three photoelectron escape depths are detected. Escape depths are on the order of 15-35 Å, which leads to an analysis depth of ~50-100 Å. Typically, 95% of the signal originates from within this depth.

Analytical Parameters

Instrument	PHI Quantum 2000
X-ray source	Monochromated AlK_α 1486.6eV
Acceptance Angle	$\pm 23^\circ$
Take-off angle	45°
Analysis area	200 μm
Charge Correction	C1s 284.8 eV
Ion Gun Conditions	Not Applicable
Sputter Rate	Not Applicable

Results and Discussion:

Survey spectra and high energy resolution spectra are found in the attached figures. Sample names are provided in the title box of each spectrum. High resolution data have the appropriate element indicated above each panel.

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C.4

Table 1 provides the atomic concentrations of the elements detected. XPS does not detect H or He. Values given are normalized to 100% using the elements detected. Detection limits are approximately 0.05 to 1.0 atomic %. Major factors affecting detection limits are the element itself (heavier elements generally have lower detection limits), interferences (can include photoelectron peaks and Auger electron peaks from other elements) and background (mainly caused by signal from electrons that have lost energy to the matrix).

Chemical state assignments for a given element have been made by consulting reference data from the literature. Chemical state assignments must be considered tentative in cases where the available reference data is limited or in cases where similar binding energies are observed for a number of different chemical states.

Non-linear least squares (NLLS) curve fitting have been applied to the C1s, Ti2p, F1s, N1s and O1s high resolution spectra to assist in possible chemical state assignment. Results of the NLLS fits are shown on the individual spectra. Please note that curve fit routines are mathematical devices, and for a given peak a number of mathematically valid results may exist.

1. The residue is a mixture of Ti, N, O, F and C (see Table 1).
2. While titanium (Ti) would be expected on the surface, the observed chemistry is complex. A significant portion of the Ti is found as TiO_2 (see Table 2) which would be expected on an exposed TiN surface that has oxidized. Other observed expected species are TiO, TiN and $\text{Ti}_x\text{O}_y\text{N}_z$. However, an unexpected high binding energy signal is detected which is characteristic of TiF_3 and/or TiF_4 .
3. High levels of fluorine (F) are detected on the surface containing the residue. The F curve fits in Table 3 show that inorganic fluoride consistent with $\text{TiF}_3/\text{TiF}_4$ are present. However, in addition to the inorganic fluorine, a second peak consistent with a fluorocarbon is also present.
4. The carbon (C) chemistry is very complex. C is found predominantly as a hydrocarbon (C-C, C-H), however, significant quantities of O-, N- and F-containing species are also present (see Table 4). The observation of CF species is consistent with the fluorocarbon signal discussed in Point #3 above. It should be stressed that other species not listed in the table may also be present. Likewise, inclusion of a species within the table does not confirm its presence. It should also be noted that some of the detected C likely originates from adventitious C. Adventitious C arises from the adsorption of atmospheric C-containing species onto sample surfaces.
5. Nitrogen (N), like Ti, is also expected on the TiN surfaces. However, as with Ti, the N chemistry is more complex than expected. N is present predominantly as TiN. However, a lower binding energy nitride is also detected (see Table 5). Based on the concentrations and the fact that no other metals are present, it would appear that this is another form of a Ti nitride, possibly with a different stoichiometry from TiN. In addition,

C.5

$Ti_xO_yN_z$ is detected as are organic N species. The organic N could be from residual photoresist. An ammonium species is detected as well, suggesting the presence of an ammonium fluoride.

6. Oxygen (O) is found on the surface as both inorganic species (e.g. TiO_2 , $Ti_xO_yN_z$) and organic species (e.g. C-O, C=O). This is shown more clearly in the O curve fits of Table 6.

Table 1: Atomic Concentrations (in %)^a

	C	N	O	F	Ti
Residue on V2	21.3	12.6	31.8	17.7	16.6

^a Normalized to 100% of the elements detected. XPS does not detect H or He.

Table 2: Titanium Chemical State (in %Ti)

	TiO, TiN	Ti ₂ O ₃ , Ti _x O _y N _z	Ti ₂ O ₃ , Ti _x O _y N _z	TiO ₂	TiF ₃ , TiF ₄
Residue on V2	11	9	7	65	8

Table 3: Fluorine Chemical State (in %F)

	Fluoride	Fluorocarbon
Residue on V2	53	47

Table 4: Carbon Chemical State (in %C)

	C-C, C-H	C-O, C-N, CH ₂ -CF ₂	C=O, CHF	O-C=O, -CF-CF ₂	CO ₃ , CF ₂ -CH ₂	CF ₃
Residue on V2	46	22	8	15	7	2

Table 5: Nitrogen Chemical State (in %N)

	Other Nitride	TiN	Ti _x O _y N _z , Organic, C≡N	Organic, C≡N	NH ₄
Residue on V2	16	35	10	24	15

Table 6: Oxygen Chemical State (in %O)

	Inorganic O	Organic O
Residue on V2	74	26